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High-Pressure Powder Diffraction Experiments using Imaging Plate at Beamline X7B

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Beamline(s): X7B

Pressure is a basic thermodynamic variable transforming matter from one state to another. However, our knowledge of pressure-induced phase transformations is still very limited compared to the vast number of temperature-dependent phases we have characterized over the past decades. This is largely due to the experimental difficulties involved in both the need for specialized equipment and the development of expertise in the use of high-pressure cells. Over the last couple of years, we have developed necessary techniques performing high pressure experiments using diamond-anvil high-pressure cells (DAC), monochromatic synchrotron X-ray and position-sensitive detector (PSD) at the beamline X7A and identified a nascent field of high-pressure crystallography, which merits a concentrated effort: pressure-dependent chemistry in nanopores.¹ Although many successful experiments were made using the medium-resolution ($\Delta d/d \approx 10^{-3}$) PSD at X7A, it was in most cases that the small-sized high-pressure sample chamber ($\sim 200 \mu\text{m}$ diameter) hampers good powder-averaging, which is crucial in performing accurate structure refinements. The imaging plate (IP) area detector at X7B can be used to avoid such problem by collecting full Debye-Scherrer rings, and any texture-related effects can be detected or corrected in resulting powder diffraction patterns. In addition, the IP will greatly reduce data collection time compared to PSD (for example, 5 min exposure using an IP compared to 3 hours scan using a PSD) although its resolution may limit the type of materials to high-symmetry systems. After some initial developments to optimize the data collection conditions, we were able to reproduce the reported bulk modulus and pressure derivative of NaCl standard within its error.² Suitable materials of interests to many chemists and physicists will be looked in the upcoming beamtimes, and we believe such efforts will extend the versatility of X7B beamline and attract a broader user community.

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References:

[1] Lee, Y.; Vogt, T.; Hriljac, J. A.; Parise, J. B.; Artioli, G. *Journal of the American Chemical Society* 2002, **124**, 5466-5475.

[2] Decker, D. L. *Journal of Applied Physics* 1971, **42**, 3239-3244.

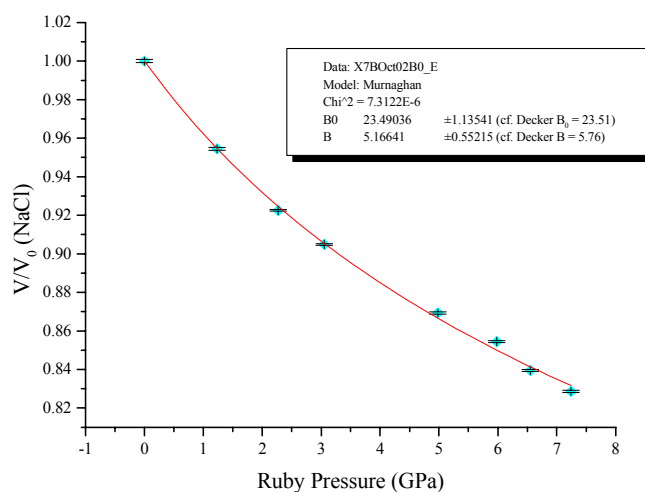
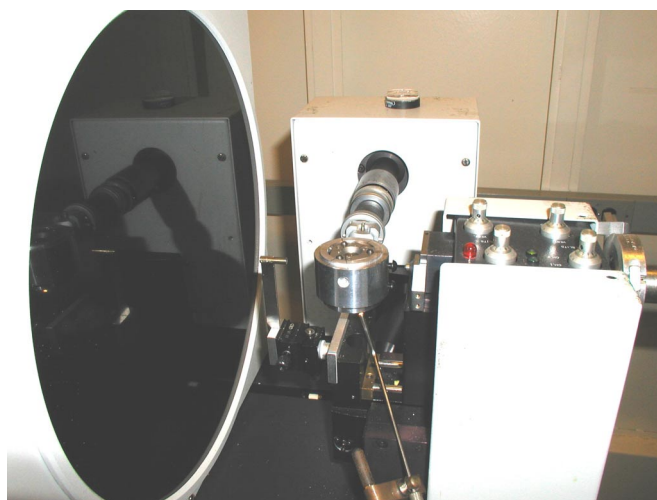


Figure. High-pressure powder diffraction setup at X7B beamline showing Mar345 on-line imaging plate and a diamond-anvil cell (left). Pressure dependence of the unit cell volume of NaCl, normalized to its ambient pressure value. Continuous line is a fit to the volume data using third-order Birch-Murnaghan equation of state.